Towards the Integral Valorization of Olive Pomace-Derived Biomasses through Biorefinery Strategies

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Supporting Information available online

Abstract

The olive oil sector generates a high quantity of biomasses every year, especially in the Mediterranean region. Olive pomace is the main one, but depending on the extraction and subsequent processing, other derived biomass by-products are generated like pâté, exhausted olive pomace, olive stone, and residual pulp. Their sustainable valorization is crucial. Therefore, this review first conceptualizes the current situation of the olive oil sector and describes these biomasses from a qualitative and quantitative point of view. Second, information on the bioactive compounds they present, the technologies used for their extraction, and examples of applications for their extracts is provided. Third, since the extraction of bioactive compounds will generate new residual biomasses, this review takes a step forward by integrating the extraction step in biorefinery cascading schemes. It also analyzes the benefits of this integration, the contribution to a circular (bio)economy, and the achievement of sustainable development goals.

Keywords: Bioactive compounds, Biorefinery, Circular economy, Olive pomace, Sustainable development goals

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1 Introduction

Olive oil is the main fat in the Mediterranean diet, and it stands out for its organoleptic, nutritional, and healthy properties [1, 2]. In 2020, the worldwide area dedicated to olive cultivation was 12.8 Mha, mainly for oil production, and it is concentrated in the Mediterranean basin [3]. According to the last estimated data in FAOSTAT, the world production of olive oil was around 3.4 million tons in 2020, and a large variation was observed in the last decade (Fig. 1a). About 90 % of the world production is concentrated in these eight countries: Spain (40.2 %), Tunisia (11.1 %), Italy (9.8 %), Greece (9.1 %), Turkey (7.1 %), Morocco (4.9 %), Syria (4.1 %), and Portugal (3.2 %) [4]. Spain is the world's leading producer and exporter of olive oil [5] and strongly influences the global trend (Fig. 1a).

The olive oil production process begins with harvesting and transporting the olives to the mill, where they undergo a cleaning process. Then, olives are milled, and the paste obtained goes through a slow stirring or mixing step, known as malaxation, to facilitate the coalescence of oil drops into larger ones and enhance oil recovery by increasing the break-up of vegetable cells [6]. Next, the olive paste is separated into two or three phases depending on the technology applied in the olive mills, which will also address the water requirements [7].

The extraction system availability depends on the country. Although there were some different values for the same country depending on the literature consulted, Fig. 1b shows a snapshot of the technology applied in the olive oil sector. While Spain mainly uses the two-phase system (~ 99 %), most of the top-producing countries use the three-phase system. The two-phase system emerged to reduce water consumption and there-

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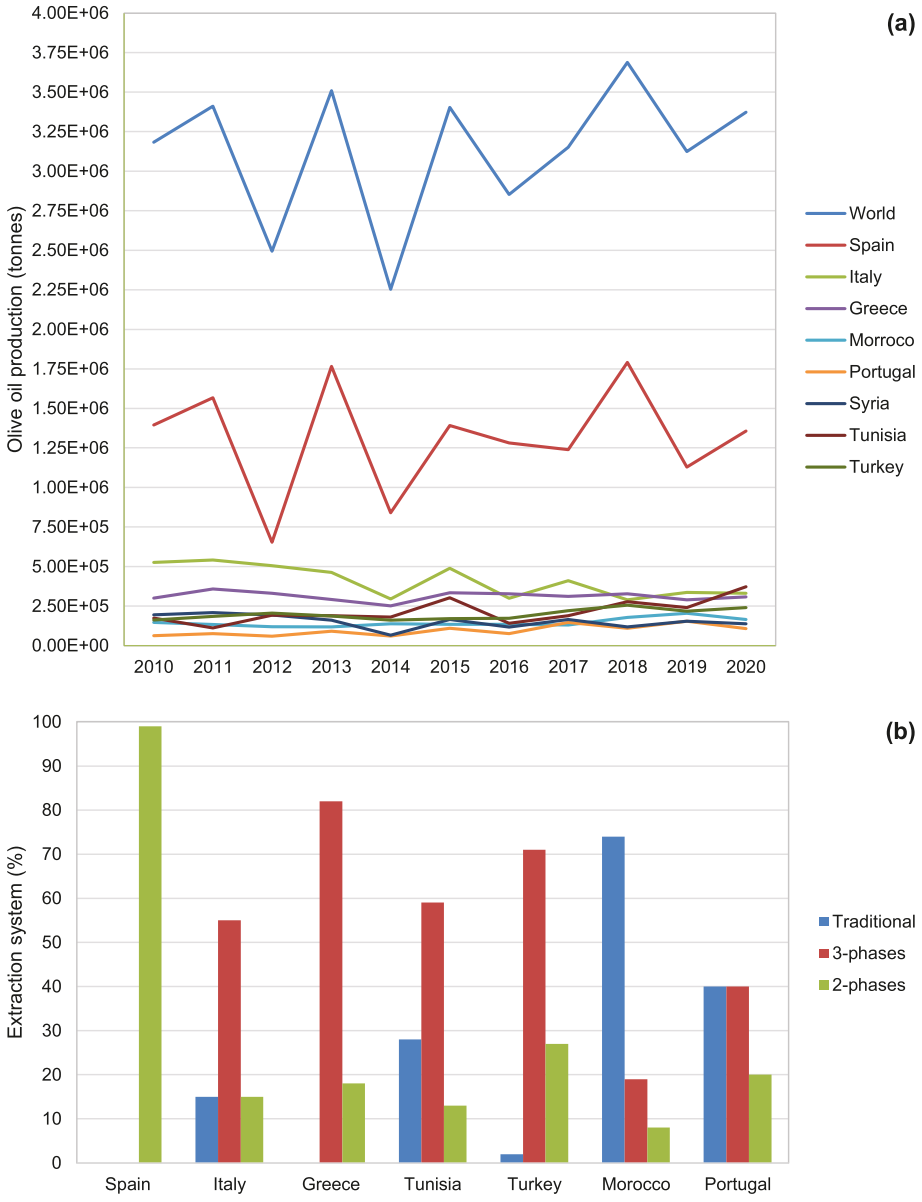


Figure 1. (a) Olive oil production and top producing countries [4], (b) type of extraction systems used for the production of olive oil according to previous studies [10, 12, 37, 150–152].

fore diminish the wastewater generated in the overall process [8]. This has resulted in more sustainable processes with CO_2 savings while providing good-quality extra virgin olive oil [6]. Some countries like Morocco are adopting this system to minimize the environmental impact [9]. Alternatively, Tunisia has increased the extraction capacity using continuous extraction systems since the late 80s, but it mainly uses the three-phase process. For example, in this country, the three-phase extraction systems represent 59 % of the production and produce about 78 % of the olive oil national volume [10].

The olive oil extraction system will strongly influence the proportion and chemical composition of the biomasses generated, including the moisture, oil, and lignocellulose content [11–13]. This fact is crucial to establish management and valorization strategies due to this sector's continuous technological changes. The lignocellulosic and thermochemical characteristics of the biomass make it a renewable source of energy and biofuel. This is important to achieve the energy transition and reduce dependence on fossil fuels [14]. Moreover, some of these biomasses are interesting bioresources to recover natural bioactive compounds [15–18] and prebiotic oligosaccharides [19], whose profile will also depend on the biomass type, among other factors [11, 15]. These biocompounds have potential applications in the pharmaceutical, food, and cosmetic sectors owing to their technological and functional properties [11, 15, 20].

Integrating the extraction of these biocompounds together with obtaining bioenergy, biofuels, and/or other value-added chemicals is a subject of current studies [21–24]. This means applying biorefinery schemes that provide value to olive pomace-derived biomass and the transition towards a circular (bio)economy based on sustainable principles and green chemistry. This transition could contribute to an increase in the profitability of the olive oil industry [25], the creation of new employment opportunities in rural areas, and the production of green energy [26]. Ultimately, it could address responsible production and consumption in the olive sector [25, 27], along with a carbon-neutral economy [3], which are some of the targets of the UN's sustainable development goals (SDGs).

In this context, this review first aims to give updates on the current situation of the olive oil sector. Second, the biomasses derived from olive pomace are described in detail, considering quantitative and qualitative aspects. Third, the types of bioactive compounds found in these biomasses are described, focusing on polyols, oligosaccharides, phenolic compounds, and triterpenic acids. Fourth, new advances in the technologies applied to recover bioactive compounds in mono-product processes and in multi-product biorefineries are reviewed and discussed, also considering sustainable aspects. Overall, this review goes one step further than what has been reviewed so far because it intends to contribute to the sustainable management of olive pomace and derived biomasses (pâté, exhausted olive pomace, residual pulp, and olive stone), not only as natural

resources of bioactive compounds but also as a basis of other bio-based products applying cascading biorefinery schemes.

To build the review, information about olive oil production was retrieved from FAOSTAT [4]. The search was then mainly focused on scientific studies published since 2015, utilizing Scopus and Google Scholar databases. The keywords were: "bioactive", "biomass", "biorefinery", "by-product", "cellulose", "characterization", "dry olive pomace", "exhausted olive pomace", "extraction", "hydroxytyrosol", "mannitol", "maslinic acid", "olive", "olive pomace", "olive pulp", "olive skin", "olive pate", "pectin", "phenolic compound", and "triterpenic acid". Specialized websites, reviews, and book chapters were also consulted when required.

2 Extraction Systems for Olive Oil Production: Where is the Olive Oil Sector Moving towards?

The conditioning of the olives before milling may vary, e.g., olives collected from soil require more intense cleaning (leaf-removal and washing) than green olives collected directly from the tree. Milling and kneading conditions are crucial factors in the production process of virgin olive oil [28], e.g., the latter may vary between 30 and 60 min with temperatures from 25 °C to 32 °C [6]. However, the extraction system will be the main factor affecting the proportion and composition of the biomasses obtained in the olive production process [11]. As an example, Fig. 2 shows representative block diagrams of the extraction of virgin olive oil and the mass balances, according to the literature. This will mainly depend on the water added to the process and the decanter type [10, 13, 29, 30]. Moreover, examples of images of olive pomace and the derived biomasses are illustrated in Fig. 3.

(a) The traditional separation system consists of a discontinuous hydraulic pressing (Fig. 2a). The paste is placed on pressing disks, where water (320–600 L/ton) is added [8].

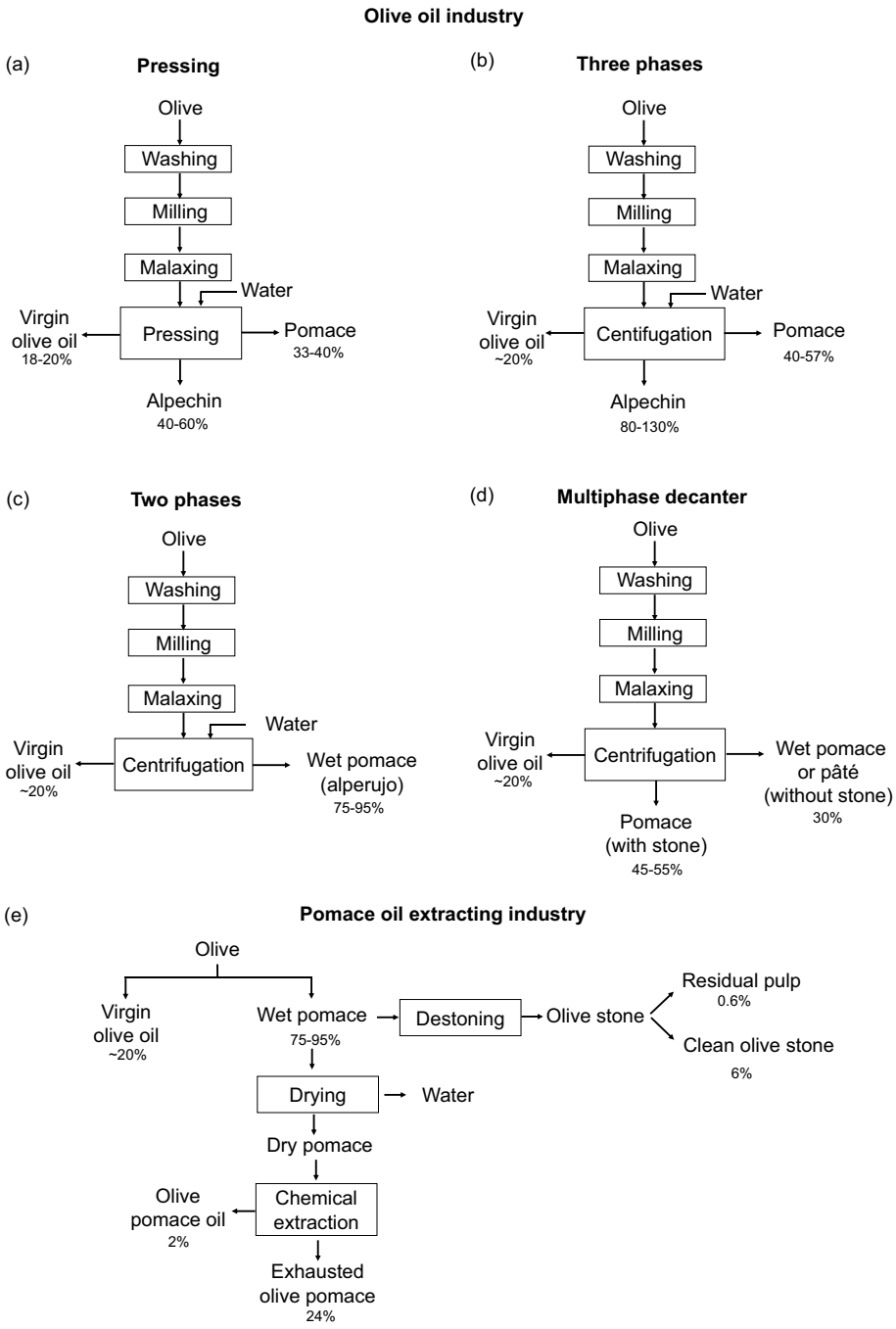


Figure 2. Diagrams of virgin olive oil and olive pomace oil production processes and the material balance expressed as percentage of 100 kg of olives, according to previous studies [13, 29, 30].

The main driving force for separation is pressure, generating a liquid fraction composed of water and oil and another solid fraction known as olive pomace [31]. Then, the oily must is separated, e.g., using vertical centrifugation [6]. Although this system is still employed, as shown in Fig. 1b, the use of continuous decanters provides high loading capacity and lower production costs [6]. In this process,

about 33–40 kg of olive pomace per 100 kg of olives is obtained (Fig. 2a).

(b) In the three- and two-phase extraction systems, a horizontal decanter is used to separate the oily phase in continuous mode through centrifugal action at about 3000 rpm [13]. In the former case, 350–700 L/ton of water are added to the paste [8]. Subsequently, it is centrifuged, thus obtaining olive oil, vegetable water (or alpechin) (80–130 kg per 100 kg olives), and olive pomace (40–57 kg/100 kg olives) (Fig. 2b).

(c) In the two-phase extraction system (Fig. 2c), the paste is centrifuged as before to separate the oil from the olive pomace, but two fractions are obtained, two-phase olive pomace (alperujo or alpeorujo), which is a mixture of alpechin and olive pomace [11]. This system allows the production of olive oil without the need to add water to the decanter, increasing the production of olive pomace (about 80 kg per 100 kg of olives), but alpechin is not generated. Water can be added to facilitate the extraction of olive oil when the humidity of the olive is low [32]. Then, the water consumption in this system is about 120–350 L/ton [8].

(d) Recently, a continuous multiphase decanter (DMF) has been introduced in some Italian and Spanish oil mills [13, 33]. It is considered an evolution of the two-phase decanter, working at 3350–3800 rpm without adding water [34]. As a difference between the aforementioned systems, DMF produces oil, an olive pomace with stone content and moisture similar to the three-phase one, and a more wet olive pomace called "pâté" (also called pâté olive cake) [35]. It generates about ~50 kg of olive pomace and 30 kg of pâté per 100 kg of olives (Fig. 2d).

Besides the aforementioned ways of extracting olive oil, another practice is to use the percolation system in the separation step, but it is not common [6]. Moreover, double extraction (two sequential centrifugation steps) of olive oil is commonly applied in some countries like Spain and Tunisia to increase profit [6, 10]. For example, a practice in Tunisia combines two-phase and three-phase extraction. The latter is applied to recover around 40–50 % of the olive oil remaining in the two-phase olive pomace [10]. A partial destoning of the two-phase olive pomace is also achieved in Spain [6], either in the oil mill or in the olive pomace oil extraction industry. This mechanical fractionation leads to generating stones or pits and a partially pitted pomace [36] whose stone content will depend on the sieve used. Some industries related to the olive sector clean the stone to improve its properties as fuel, generating another by-product that is called residual olive pulp or the residual fraction from olive stone cleaning [12, 15]. Since it is rich in the skin [17], this biomass is also known as residual olive skin (as an example, see Fig. 3).

Currently, the main uses of three- and two-phase pomace is to recover the residual olive oil with organic solvents such as hexane (Fig. 2e), but selling olive pomace has poor profitability for the oil industry [37]. The olive pomace extracting industries have been adapting to the innovation in the former sector, but not without difficulties. Among these problems are the following: The applied solvent is not "green", olive pomace contains low oil content (up to 3.5 %), especially, when double centrifugation and two-phase decanters are applied. Moreover, intensive drying of the pomace is required for extraction, increasing costs and uncertainty when it depends on fossil fuels. Overall, this could be related to the fact that olive pomace oil extraction plants have been dismissed in some countries, while in other countries like the USA this industry has not emerged [13]. Nevertheless, some industries use the dry and exhausted olive pomace (also called de-oiled and defatted olive pomace) obtained in the extraction process as a fuel resource to increase profitability. Other products can be obtained in the refining process of the olive pomace oil, like fatty acids and refining pastes [38].

Innovative approaches like DMF can support this industry since the olive pomace produced has low water content and much higher energy because it contains all the woody parts of the stone [13]. Other trends move towards three-phase extraction and the forced evaporation of alpechin, e.g., using an Alfa-Flash plate flash evaporator [39]. As far as we know, these technologies are only marginally applied.

3 Olive Pomace and Derived Biomass: Description and Chemical Composition

As Fig. 2 and Tab. S1 (Supporting Information) demonstrate, the extraction technologies and the conditions applied in the olive oil production process will lead to different waste products, varying in the proportion and composition [11, 12, 40, 41]. The main by-products obtained are olive pomace (from pressing, two-phase, and three-phase systems) and, in the case of the DMF system, olive pomace (with stone) and pâté (without stone) (Fig. 3). For example, the oil content may vary between 3 % and 9 % in the traditional pomace, 2.5 % and 6 % in the three-phase pomace, and 2 % and 3.5 % in the two-

Figure 3. Images of olive pomace and derived biomasses.

phase pomace, on a fresh basis [1]. Pâté could increase the oil content up to 10.3 % on a fresh weight basis [35]. These values are higher if considered on a dry weight basis (fat content in Tab. S1). The moisture content can reach values of about 54 %, 79 %, and 86 % in three- and two-phase pomace and pâté, respectively (Tab. S1). The olive pomace from pressing the moisture could be up to 28 %.

In addition, Tab. S1 shows the chemical composition (extractives, cellulose, hemicellulose, lignin, protein, and fat content) of different olive pomace-derived biomasses, which is crucial to consider in biorefinery cascading schemes in order to select the best valorization route in each case. The olive cultivar, farming practices, and fruit ripening can also contribute to variation in the chemical composition of these biomasses [40–42]. This explains, at least in part, the observed variation of the data within the same biomass.

3.1 Olive Pomace and Pâté

Olive pomace is made up of a solid fraction (skin, pulp, and stone), oil, and vegetation water [11]. As commented before, the moisture and oil contents mainly depend on the extraction system, but all olive pomaces have a lignocellulosic composition and high extractives content (Tab. S1).

Olive pomace is a rich source of lignin, which is mainly related to the stone. It may account for up to ~ 40 % (dry basis), although a large variation exists between the data reported in the literature. Other major components are cellulose and hemicellulose, while its content of fat and protein is generally lower (Tab. S1). Certain variations in these contents have been found in olive pomace of different origins [40], which can be attributed to the olive cultivar, ripening degree, and processing practices [40, 42].

The main application of olive pomace is the production of olive pomace oil, as commented in Sect. 1. Although it has also been used for animal feed [43], lignin contributes to poor digestibility [44]. Nonetheless, olive pomace has also been incorporated into food (biscuits, snacks, oils, pasta, meat, etc.) and feed, increasing the phenolic and unsaturated fatty acid contents, fiber content, and the oxidative stability of these products, among other effects [45]. Other applications have been found for olive pomace such as a fertilizer and for energy purposes (production of methane and syngas) [45, 46] and as an additive in materials to improve properties like resistance to oxidation-induced cracking [47] and reduce thermal conductivity [48]. Moreover, it can be used as a source of lignin [41], phenolic compounds [49, 50], and pectins [51], which are objects of study in Sect. 4.

Pâté is a semi-solid, pitted by-product obtained from the new two-phase decanter DMF [12] (Fig. 2d). It consists of vegetation water, olive pulp, olive skin, and residual olive oil. On a dry basis, it has a low lignin content (about 9 %) compared to the rest of the studied biomasses, but high-fat content (up to 30 %) on a dry weight basis (Tab. S1).

Because this biomass has a low lignin content, its applicability in the food sector itself is promising. It is even suitable for formulating new functional foods, nutraceuticals, and chicken feed [30, 35, 52, 53]. Pâté also presents a typical olive oil fatty acid profile [35] and high content of phenolic compounds [54].

3.2 Exhausted Olive Pomace, Olive Stone, and Residual Pulp

Exhausted olive pomace is the dry biomass resulting from drying and extracting the residual oil in olive pomace oil-extracting industries (Fig. 2e). It has a low moisture content (Tab. S1), and it is made up of skin (15–30 %), stone (30–45 %), and pulp (30–50 %) [55]. Nonetheless, the stone fraction will depend on the grade of destoning of the olive pomace. This biomass is chemically composed of cellulose (~ 8–23 %), hemicellulose (~ 9–24 %), lignin (~ 21–42 %), and a high content of extractives (higher than 30 %) (Tab. S1).

Currently, this by-product is used locally as a biofuel for generating heat and electricity, with a caloric power of 3200–3600 kcal kg^{-1} [6]. During its combustion, particles that are harmful to human health and the environment are emitted [56]. This fact has prompted looking for new valorization ways, e.g., to obtain fermentable sugars [22, 23, 55], ethanol [57], xylitol [58], polygalacturonase and fatty acids [59], proteins [24], lignin, and bioactive compounds [22, 23, 55].

Olive stone is mainly recovered from two-phase olive pomace by centrifugation and sieving, and those generated in the oil mill have calibers between 1 mm and 1.6 mm. This is an advantage compared to other olive by-products since it is easy to store. Olive stone represents around 18–25 % of the weight of the olive, although about up to 9 % is recovered, and the rest remains in the olive pomace to make the drying and chemical extraction step easier (Fig. 2e) [60].

Its main components are cellulose (~ 14–21 %), hemicellulose (19–30 %), and lignin (36–42 %), while its content of extractives is low compared to the aforementioned biomass. These values can vary depending on the geographical location, olive cultivar, and the presence of traces of pulp and skin [61] (Tab. S1). Owing to its high calorific power (4000–4200 kcal kg^{-1}), this biomass is used as a biofuel in domestic heating systems or small industrial boilers [6, 62], providing new income in the oil mills and associated industries [6]. Moreover, its combustion produces a very low quantity of ash, smoke, and gases, with low sulfur and nitrogen content, compared to exhausted olive pomace [6]. Other possibilities of valorization could be through obtaining antioxidants [36], sugars and derivatives like ethanol [56, 63], xylitol, furfural [64], and lignin [62].

The residual olive pulp or skin consists mainly of the remains of olive pulp and skin [15], but it presents a high content of lignin (~ 32 %), suggesting the presence of stone. Other components are extractives (~ 26 %), cellulose (~ 12 %), and hemicellulose (~ 14 %) (Tab. S1). This biomass contains a high proportion of extractable compounds soluble in ethanol (18 %) compared to exhausted olive pomace (4 %) and stone (2 %) [15, 64]. Currently, it does not have any industrial application [15], although local ranchers use it as feed owing to its fat content [43]. Recent studies suggest a potential application to obtain bioactive compounds, specifically triterpenic acids [17] and phenolic compounds [15].

4 Bioactive Compounds in Olive Pomace and Derived Biomasses

This work refers to bioactive compounds, such as those naturally present in foods and derived by-products/wastes, that do not necessarily have nutritional value but can produce beneficial effects for health. Olive pomace-derived biomasses contain a wide range of bioactive compounds in the extractive fraction. Fig. 4 shows examples of their chemical structure. These can be extracted with solvents, as this review will detail in Sect. 5. Their qualitative and quantitative composition will depend on the biomass type [15]. Other crucial factors are the olive cultivar, fruit collection mode, and olive oil extraction technology, among others [11, 42].

This review has focused on sugar derivatives, phenolic compounds, and triterpenic acids owing to the current industrial interest. Moreover, oligosaccharides are another interesting fraction from these olive sources, which can be obtained by treatments with mild conditions. However, these components are generally in low concentrations in biomass, and thus the integration of production routes within biorefinery strategies is desirable to promote a circular (bio)economy model, as discussed in Sect. 6. Moreover, if other routes are performed, like thermochemical ones, these interesting compounds are lost with no revenue. In this sense, the tip of the biomass valorization pyramid is occupied by compounds with pharmacological properties or fine chemicals to improve health and lifestyle, while bioenergy and composting are the least-priority options (i.e., the basis) [26]. Therefore, the recovery of these bio-based molecules is interesting, but with biorefinery thinking it could be more advantageous as it means integral utilization of the biomass.

4.1 Sugar Derivatives: Polyols, Oligosaccharides, and Pectins

4.1.1 Polyols

Polyols (or alditols or sugar alcohols) are no noncyclic hydrogenated carbohydrates where the carbonyl group (aldehyde or ketone) is reduced to a hydroxyl group (e.g., erythritol, mannitol, xylitol, sorbitol, etc.). This type of compounds are sweet-tasting compounds with low digestibility. They are used as low-calorie sweeteners [65].

Particularly, mannitol (Fig. 4) is the main polyol found in the by-products of the olive industry, although xylitol has also been reported in olive leaves [20]. Mannitol and xylitol are C6 and C5 polyols presenting the molecular formulas $C_6H_{14}O_6$ and $C_5H_{12}O_5$, respectively. Besides its sweet taste, mannitol occurs as a white, odorless substance and imparts a cooling sensation in the mouth [66]. Mannitol can also be applied as an antioxidant preservative and reducer of sugar crystallization in the food and cosmetics industries [67]. Moreover, it presents clinical applications as an osmotic diuretic drug [68]. It has nonhygroscopic properties, making it a potential excipient for moisture-sensitive drugs [66]. Also, it has the potential to be applied for the treatment of diseases such as Parkinson's disease, cystic fibrosis, migraines, and renal insufficiency [22].

4.1.2 Xylo-Oligosaccharides

Hemicellulose is a group of cell wall polysaccharides that have a low degree of polymerization and low crystallinity, which makes them soluble in diluted inorganic acids and alkalis at high temperatures [69]. The main chain of hemicellulose can be made up of a single sugar, homopolymer (e.g., xylan), or two or more sugars, heteropolymer (e.g., glucomannan) [70]. In particular, xylans are one of the main classes of hemicelluloses that accumulate in secondary cell walls, but they are also components of the primary walls of dicots, which includes the olive tree [71]. The type and content of hemicellulose vary depending on biomass type (Tab. S1), but in olive pomace and derived biomass, xylose is the main carbohydrate that makes up this polymer (xylan), followed by galactose and mannose. The percentage of xylans is about 8.0 % in two-phase olive pomace, 9.5 % in exhausted olive pomace [14, 15, 56], 23.5 % in olive stone [36, 56], and 13.5 % in residual pulp [15].

Oligosaccharides are carbohydrates consisting of short chains of monosaccharide units (usually 3–10 units) and are soluble in water. Oligosaccharides from olive by-products can be obtained from hemicellulose through steam treatment or autohydrolysis [62]. They are not cariogenic, and they have prebiotic properties. Therefore, they present great potential for formulating functional foods and pharmaceutical products [72]. The hemicellulose fraction of lignocellulosic biomass is known to be a rich source of oligosaccharide production [73], most notably xylo-oligosaccharides (XOS) that derive from xylans [42]. In this type of oligomers, *D*-xylose residues are linked via β-1,4-glycosidic bonds [74], e.g., Fig. 4. Moreover, xylans can also present side groups like acetyl and uronic acids [74].

4.1.3 Pectins

Pectins are complex galacturonic acid oligomers (Fig. 4) present in the middle lamella and in the primary cell wall of plants that provide firmness to plant tissue. It is a polymer with linear structure in which few hundred to thousand galacturonic acid units are linked via α-1,4-glycosidic bond forming the backbone, which can be substituted with other neutral sugars (e.g., rhamnose, galactose, mannose, glucose, and xylose). Methyl esterification of galacturonic acid also occurs [75]. Due to suitable technological (e.g., emulsifying activity and stability) and functional properties (e.g., prebiotic, anti-inflammatory, etc.), pectins have many applications as (techno)functional ingredients in the food, cosmetic, and pharmaceutical industries [51, 76, 77].

Olive pomace has been described as a rich source of pectins [75, 78], with some properties similar to apple pectins, to be applied in the food and pharmaceutical industries [49–51]. Nonetheless, olive pomace pectin seems to have a higher degree of methyl-esterification, acetylation, and total neutral sugar content than apple pectin [75]. Extracts rich in phenolic compounds associated with pectins (called pectoliv extracts) have also been recently produced from two-phase olive pomace. These pectin-based products have about 50 % of sugar residues in the form of uronic acids, while also containing neutral sug-

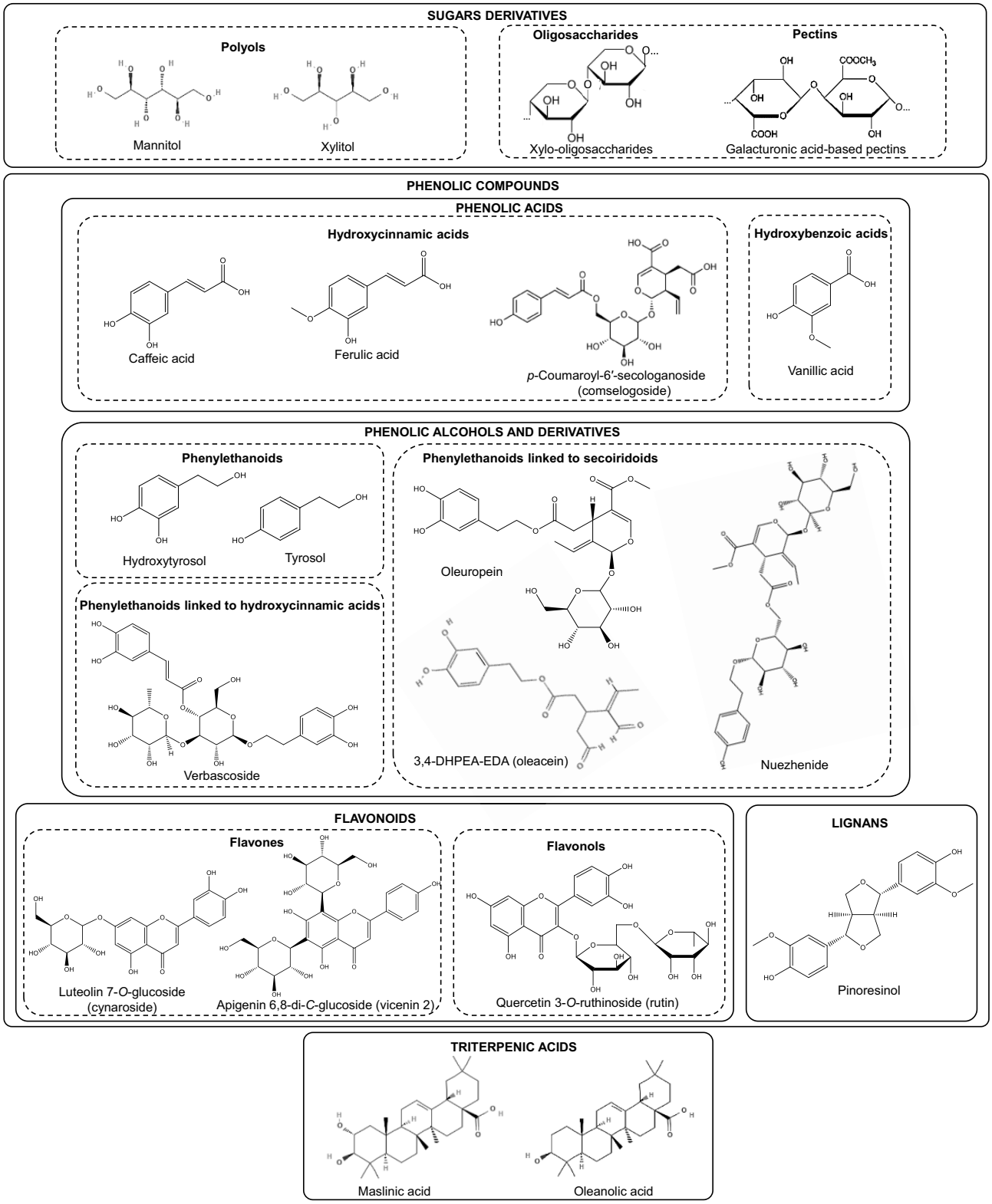


Figure 4. Chemical structure examples of bioactive compounds found in olive pomace and derived biomasses.

ars, mainly xylose and galactose, and phenolic compounds (6–8 wt %). Moreover, these extracts have shown bioactive potential against cancerous cells [79].

4.2 Phenolic Compounds

Phenolic compounds are one of the most important groups of phytochemicals or plant secondary metabolites [80]. They have numerous applications in the food and pharmaceutical industry [49, 50]. They are composed of an aromatic ring of six carbon atoms normally attached to at least one hydroxyl group (–OH) and other functional groups. For example, by O-bonds, phenolic compounds are attached to monosaccharides like glucose and rhamnose, disaccharides like rutinose, terpenes like secoiridoids, organic acids like quinic acid in chlorogenic acid, etc. [15, 81]. Also, they can be attached to sugars through C-glycosidic bonds [82]. Examples of phenolic compounds occurring in olive biomasses with these structures are given in Fig. 4.

Based on the number of aromatic rings they contain and their chemical structure, olive phenolic compounds can be classified as simple phenols, phenolic alcohols or phenylethanoids, phenolic acids, flavonoids, tannins, and lignans [83]. Nonetheless, phenylethanoids can be simple forms like hydroxytyrosol (HT) and its glucoside or more complex forms attached to secoiridoids like oleuropein and derivatives. This can also be observed for hydroxycinnamic acids. For example, caffeic acid may appear solely or linked to sugars (e.g., glucose) and organic acids (e.g., chlorogenic acid) but also coupled to phenyethanoids like verbascoside and secoiridoids like caffeoyl-6'-secologanoside [11, 15] (Fig. 4).

Besides the antioxidant capacity of olive phenolic compounds in biological and food systems [84, 85], olive phenolic compounds present a wide range of pharmacological properties, as reviewed in [12, 86]. As an example, Tab. S2 shows some in-vivo and clinical studies on HT, one of the main interesting olive phenolic compounds found in olive pomace [49] and derived biomasses like pâté [87] and exhausted olive pomace [88]. For example, HT protects against lipid oxidation and prevents cardiovascular diseases [89]. Combined with vitamin E, it improves steatosis and hypertriglyceridemia in children [90]. HT and HT-rich extracts could be useful to formulate functional ingredients based on these clinical studies. This compound is also considered a novel food ingredient for food preservation that can be added to vegetable and fish oils and fat spreads [91]. This means that there is currently great interest in obtaining HT in different ways [92], but through extraction, natural resources like the object of this review can be repurposed.

4.3 Triterpenic Acids

Terpenes are natural secondary metabolites found in various animal and plant species [93]. These compounds are hydrocarbons classified by the number of isoprene units (C_5H_8), the most common being mono-, sesqui-, di-, and triterpenes (C_{10}, C_{15}, C_{20}, C_{30}, respectively).

Olive biomasses contain monoterpenes and triterpenes. The former can be found free or bound to phenolic compounds, as commented in Sect. 4.2. Triterpenes are found as free forms in olive biomasses and have raised great interest in recent years. Based on their structure, triterpenes can be divided into linear, tetracyclic, and pentacyclic [94]. One of the most important classes of olive triterpenes belongs to this last type, highlighting triterpenic acids and triterpenic alcohols. In particular, maslinic acid and oleanolic acid are the main triterpenic acids present in olive pomace and some derived biomasses like exhausted olive pomace [22] and residual pulp [17, 95]. These compounds have anti-inflammatory, antihyperlipidemic, antitumor, anti-arthritis, and hepatoprotective properties, being valuable molecules for the pharmaceutical, nutraeutical, and cosmetic industries [18, 96, 97]. Extracts rich in triterpenic acids can also be suitable to formulate functional feed [98].

4.4 Examples of Industrial Applications of Olive Bioactive Compounds

As detailed in Sect. 3, olive pomace and some derived biomass have been applied as food, feed, and material ingredients. Extracts from these renewable resources can also find applications in these sectors, depending on the compound type. The extraction will reduce the complexity of the original biomass, concentrating the compounds of interest. At the same time, the solid fraction will be enriched in non-nutritive components such as lignin, cellulose, and hemicellulose [85]. Tab. S3 shows some examples of the applications of extracts containing bioactive compounds based on recent studies.

5 Extraction Technologies and Conditions Applied to Recover Bioactive Compounds from Olive Pomace and Derived Biomasses

5.1 Brief Description of the Main Technologies and Solvent Trends

5.1.1 Technologies Applied for Extraction

The extraction conditions applied to recover bioactive compounds will determine the selectivity, co-extraction phenomenon, yield, and purity of the extracts, depending on the biomass type [15, 99, 100]. Considering the bioactive compounds' physicochemical properties and their profiles in olive pomace-derived biomasses, the type of compounds extracted and their purity will vary. For example, using the same extraction conditions, between 3 (exhausted olive pomace) and 15 times (residual pulp) depending on the biomass type, Contreras et al. [15] obtained an extract enriched in bioactive compounds.

Although more reviewed studies performed extraction at a laboratory scale, their results can be the basis to select the best technology in terms of yield, purity, bioactive compound profile, and energy requirements. In these works, extraction technologies include conventional solid-liquid extraction meth-

ods such as Soxhlet extraction, maceration, aided or not by heat conduction, and emerging technologies that comply with the principles of "green" chemistry or "green" technologies. "Green" technologies offer a sustainable and eco-friendly approach to obtaining desired bioactive compounds through reduced extraction time and energy consumption [18]. These technologies are expected to contribute to reducing greenhouse gas emissions, mitigating climate change, and preserving natural resources for future generations. These technologies are also known as "process intensification technologies", although the definition of this term is more general and encompasses equipment and methods "that can provide dramatic improvements in decreasing equipment-size/production-capacity ratio, energy consumption, and waste generation" [101, 102].

Homogenate-assisted extraction (HAE) is an emerging alternative to conventional homogenization for extracting bioactive compounds in a short time and at low temperatures. HAE uses mechanical shearing at high speed as the principal mechanism of action [103]. Ultrasound-assisted extraction (UAE) is a non-thermal technology based on the incidence of ultrasonic waves with frequencies between 20 and 100 kHz, producing different effects such as cavitation, vibration, agitation, and pressure on the biomass [104, 105]. High hydrostatic pressure-assisted extraction (HHPAE) is also a non-thermal extraction technology based on the use of high pressure as a driving force to favor solubility and mass transfer of the desirable compounds into the solvent in a short time [106].

Supercritical fluid extraction (SFE) is based on using supercritical fluids, i.e., solvents that behave like a heavy liquid with the penetration capacity of a gas that diffuses through solid materials with improved transport properties [107]. SFE has numerous advantages, such as high selectivity, environmental safety, and reduced consumption of organic solvents [108]. Supercritical CO_2 is commonly used for extraction at supercritical conditions, i.e., above its critical point (~ 31 °C and ~ 74 bar). Depending on the purpose, it can act as the sole solvent or be aided by cosolvents like ethanol to modify its solubilization properties [109]. As a solvent, supercritical CO_2 has advantages for the recovery of labile or easily oxidizable bioactive compounds [110].

Microwave-assisted extraction (MAE) is based on the use of microwaves with frequencies between 0.3 and 30 GHz, making it possible to heat a matrix externally and internally [111]. In MAE, the solvent should be able to absorb and transfer microwave energy, while UAE requires solvents with low viscosity [8]. Although ultrasound can reach temperatures of 75 °C at the end of the process, caused by the ultrasonic waves themselves, the process temperature required is lower than using MAE or conventional extraction. Hence, UAE or other non-thermal technologies can be useful in the case of extracting thermolabile bioactive compounds [88].

Pressurized liquid extraction (PLE) uses liquid solvents at elevated temperatures and pressure to improve the speed and efficiency of the extraction [112]. Using high-polarity solvents such as water and ethanol at temperatures above their atmospheric boiling point improves solubility and mass transfer properties [113]. PLE generally requires shorter extraction times than MAE, but some undesirable compounds like HMF can be generated under the more aggressive conditions [22]. Therefore, the impact of the temperature should be carefully studied. High temperatures may improve extraction but can also favor degradation of the compounds of interest [20].

5.1.2 Solvents

For an environmentally sustainable extraction process, selecting the solvent is also a crucial factor, especially when looking for alternatives to conventional, toxic organic solvents [42]. Water or steam, ethanolic water solutions, and supercritical CO_2 are currently affordable and greener than solvents of fossil origin. For example, ethanol can be obtained from renewable resources in biorefineries, while CO_2 is the object of novel carbon capture technologies to reduce its atmospheric levels [3]. It is even more interesting if extraction is integrated with other biomass conversion routes, like bioethanol production, to favor its utilization for extraction [114]. Extraction and conversion of biomass can also be coupled with carbon capture technologies for utilizing CO_2 as a solvent [115]. This technology is deployed, but large-scale operation is still scarce, among other barriers [116].

Natural deep eutectic solvents (NADES) also align with the "green chemistry" principles. For example, they present low toxicity and flammability [117]. This type of solvent is based on the use of a hydrogen bond acceptor (e.g., choline chloride) and hydrogen bond donors (e.g., organic acids) that form an eutectic mixture. A recent review suggests that NADES have the potential to be used for the sustainable recovery of bioactive compounds from biomass due to their natural origin and compatibility with food, pharmaceutical, and cosmetic uses [8]. Nonetheless, to really be "green", the sustainable and competitive production of this type of solvent from renewable sources should be warranted. An other barrier could be the NADES cost and their efficient recycling to separate the target compound. However, some NADES used for extraction can be suitable as a vehicle of the bioactive compounds for direct application in food, cosmetics, and drugs [118]. For example, a recent study suggests the possibility of using an NADES formulation to encapsulate β-carotene in whey protein concentrate for industrial applications [119].

5.2 Application Examples for Obtaining Sugar Derivatives

Besides olive leaves, mannitol is present in olive pomace and exhausted olive pomace in amounts comparable to olive leaves. Alternatively, the mannitol content of olive stone is almost insignificant compared to the former (e.g., 180 times lower) [56] (Tab. 1). The concentration of mannitol in the olive pomace may vary depending on the olive ripeness, as the study of Ribeiro et al. [120] suggests. Therefore, besides the biomass type and the extraction technology, ripeness is a factor to be considered.

Concerning extraction technologies applied for mannitol recovery, there are several options (Tab. 1). This compound has been obtained in the liquid fraction from two-phase pomace recovered by centrifugation at 4000 rpm. This gave an extract

rich in mannitol (up to 324 mg g^{-1} of extract), although the yield of solids was low [67, 120].

Owing to the polar character of mannitol, its extraction from exhausted olive pomace has been studied using water as the extraction agent and applying various technologies. For example, using UAE and MAE, mannitol was solubilized at up to 50.9 mg g^{-1} biomass and 46.7 mg g^{-1} biomass, respectively, in a short time (16 min) [22, 88] and with better efficiency than using Soxhlet extraction aided by heat conduction [55, 56] (Tab. 1). In MAE, both temperature and time strongly influenced the extraction efficiency of mannitol, favoring high temperatures (up to 100 °C) but using short extraction times to avoid degradation [22]. While in UAE, the main parameter affecting extraction was the amplitude, which is related to the ultrasound intensity, and also by time [88, 121]. UAE and MAE provoked alteration of the structure, facilitating the mass transfer. For example, MAE generated large holes in the exhausted olive pomace [22], and UAE addressed erosion and the frayed appearance of the biomass surface [88].

Concerning sugar oligomers, Ribeiro et al. [67] showed that part of the pectin content of two-phase olive pomace is recovered with mannitol by centrifugation (Tab. 1), while another part is retained in a pulp-rich fraction. Therefore, a small part of these components can simply be recovered by centrifugation. Other selective strategies have been published based on using steam, as this type of treatment is considered an economically viable and environmentally friendly alternative to obtain extracts rich in sugars, oligosaccharides, and other types of bioproducts [122]. Lama-Muñoz et al. [76] carried out this type of hydrothermal treatment (170 °C for 15 min) at a pilot scale fol-

Table 1. Technologies and conditions applied to obtain mannitol and oligosaccharides from olive pomace and derived biomasses.

Extraction technique and conditions	Solvent	Yield of the compounds	Ref.
Two-phase olive pomace - mannitol			
Centrifugation (10 000 × *g*, 10 min)		141.0 mg g^{-1} a)	[120]
Centrifugation (4000 rpm, 10 min)		211.0–324.0 mg g^{-1} a)	[67]
Exhausted olive pomace – mannitol			
Soxhlet extraction (2 % w/v, 12 h)	Water	36.0 mg g^{-1} b)	[56]
Extraction aided by heating (15 % w/v, 100 °C, 30 min)	Water	39.0 mg g^{-1} b)	[55]
Extraction aided by heating (10 % w/v, 85 °C, 90 min, agitated)	Water	45.0 mg g^{-1} b)	[23]
Probe-UAE (11.5 % w/v, 80 % amplitude, 16 min)	Water	50.9 mg g^{-1} b)	[88]
MAE (12 % w/v, 100 °C, 16 min)	Water	46.7 mg g^{-1} b)	[22]
Olive stones – mannitol			
Soxhlet extraction (2 % w/v, 12 h)	Water	0.2 mg g^{-1} b)	[56]
Two-phase olive pomace – oligosaccharides			
Centrifugation (4000 rpm, 10 min)		6.9–13.3 mg pectins g^{-1} a)	[67]
Saturated steam treatment (170 °C, 15 min) + acid hydrolysis (2 N trifluoroacetic acid, 121 °C, 2 h)	Saturated steam	364.0 mg pectic polymers g^{-1} a) (>10 000 Da)	[76]
		126.0 mg XOS + xyloglucan g^{-1} a) (1000–3000 Da)	
Saturated steam treatment (160 °C, 30 min) + purification	Saturated steam	423.2 mg uronic acid g^{-1} a)	[51]
Saturated steam treatment (160 °C, 30 min) + purification	Saturated steam	386.7 mg uronic acid g^{-1} a)	[77]
Exhausted olive pomace – oligosaccharides			
LHW (3 % w/v, 230 °C, 0 min)	Water	78.0 mg XOS g^{-1} b)	[72]
LHW (10 % w/v, 130 °C, 30 min)	Water	Solubilization of 65 % of xylans	[62]
Olive stones – oligosaccharides			
LHW (190 °C, 5 min)	Water	147.0 mg XOS g^{-1} a)	[123]
LHW (3 % w/v, 230 °C, 0 min)	Water	100 mg XOS g^{-1} b)	[72]
LHW (10 % w/v, 130 °C, 30 min)	Water	Solubilization of 65 % of xylans	[62]

LHW: liquid hot water; MAE: microwave-assisted extraction; UAE: ultrasound-assisted extraction; XOS: xylo-oligosaccharides. a)Referred to extract (or liquid fraction) dry weight. b)Referred to biomass dry weight. c)Referred to wet biomass.

lowed by purification steps (liquid-liquid extraction with ethyl acetate and ultrafiltration) and acid hydrolysis to obtain XOS and pectic oligosaccharides (Tab. 1).

Other studies have used similar strategies based on saturated steam (160 °C, 30 min) to obtain an extract rich in pectins from two-phase olive pomace [51, 77]. Both studies allowed recovery of a similar amount of uronic acids, the main component of the pectins (Tab. 1). Rubio-Senent et al. [51] showed some differences in galacturonic acid content (Fig. 4), depending on the steam treatment and purification strategy. Autohydrolysis or liquid hot water treatment (LHW) has also been applied at different temperatures and extraction times to obtain XOS (Tab. 1). The effect of this treatment on both types of biomasses is similar, and the studies suggested that XOS recovery is favored at low temperatures and long extraction times [62, 72, 123]. Time and temperature are interrelated factors, so for optimal oligosaccharide production it is necessary to establish a balance between both variables [124].

5.3 Application Examples for Extracting Phenolic Compounds

Phenolic compounds have been characterized in olive pomace [49, 125], pâté [53, 87], exhausted olive pomace [56], olive stone [126], and residual pulp [67]. Currently, there is no general procedure for the extraction of phenolic compounds. It is necessary to establish how the operational factors affect the recovery of these compounds, taking into account the type of sample, the specific compound to be extracted, and the technology used for extraction [80]. For comparison, Tab. 2 shows the technology and extraction conditions applied to olive pomace and derived biomasses to recover phenolic compounds and the main phenolic compounds quantified in each case.

The extraction of phenolic compounds from olive pomace has been carried out using both conventional techniques and green technologies [49, 50]. For example, Pavez et al. [50] showed that the phenolic content, including HT, was higher in a shorter time using PLE than maceration (Tab. 2). Chanioti and Tzia [49] compared the extraction of phenolic compounds by several new technologies, UAE, MAE, HAE, and HHPAE, using water, 70 % ethanol, and NADES as solvents. All the conditions applied allowed the extraction of HT, oleuropein, vanillin, rutin, and luteolin. The highest efficiency was found when HAE and NADES (particularly formed by choline chloride and citric acid) were combined. The conditions are given in Tab. 2. In an other work, HT, caffeic acid, *p*-coumaric acid, rutin, oleuropein, and luteolin were more efficiently recovered from two-phase pomace using MAE and ethanol (50 vol %) compared to UAE and PLE using the same solvent [111].

Green technologies generally yield comparable results to Soxhlet extraction and maceration for the recovery of olive phenolic compounds when using a similar solvent, but in a shorter timeframe [20, 50]. Regarding thermal-based technologies like MAE and PLE, the increase in temperature enhances the solvent's penetration into the plant matrix, promotes the solubility of the target compounds, and ultimately accelerates the transfer of mass [20].

Cavitation will affect UAE extraction [18], in HAE the hydrostatic pressure governs the penetration of the solvent into the biomass, while in HAE the pulverization of the biomass with mechanical shear force, mixing, and fluid cutting action will be determinant for extraction [49]. The efficacy also depends on the biomass waste, e.g., while MAE showed higher efficiency for the extraction of olive pomace phenolic compounds than UAE and PLE, PLE was better for winery waste using the same conditions [111]. In this regard, mass transfer kinetic and degradation behavior will ultimately depend on the bioactive compound type present in the biomass [18, 20].

However, other factors than the yield of phenolic compounds should be considered to address multiple perspectives like energy consumption, costs, and sustainability. For example, Xie et al. [18] compared conventional solvent extraction, UAE, and MAE for the extraction of HT and triterpenic acids from two-phase olive pomace, regarding yield, energy consumption, and carbon emission. Their results showed that UAE was highly efficient for recovering these compounds in terms of yield and energy, explained by the fact that UAE shortened the

Table 2. Technologies and conditions applied to obtain phenolic compounds from olive pomace and derived biomasses.

Extraction method	Extraction solvent	Main phenolic compounds	Ref.
Three-phase olive pomace			
Maceration (25 % w/v, 120 min)	80 % methanol	Oleuropein (0.02 mg g^{-1} a))	[50]
PLE (14.7 % w/v, 136.5 °C, 20 min, 103 bar)	52 % ethanol	HT (0.07 mg g^{-1} a));	[50]
		luteolin (0.2 mg g^{-1} a));	
		oleuropein (0.1 mg g^{-1} a))	
HAE (8 % w/v, 60 °C, 30 min, 12 000 rpm)	NADES (choline chloride:citric acid, 1:2, v/v)	HT (1.1–6.1 mg g^{-1} a);	[49]
		oleuropein (7.2–12.9 mg g^{-1} a));	
		vainillin (0.06–0.56 mg g^{-1} a));	
		rutin (0.9–1.7 mg g^{-1} a));	
		luteolin (0.04–0.12 mg g^{-1} a))	

Table 2. Continued.

Extraction method	Extraction solvent	Main phenolic compounds	Ref.
UAE (8 % w/v, 60 °C, 280 W, 60 kHz)	NADES (choline chloride:maltose, 1:2, v/v)	HT (1.1–1.4 mg g^{-1} a));	[49]
		oleuropein (0.0–0.9 mg g^{-1} a));	
		vainillin (0.02–0.1 mg g^{-1} a));	
		rutin (0.2– 0.4 mg g^{-1} a));	
		luteolin (0.04–0.10 mg g^{-1} a))	
MAE (8 % w/v, 60 °C, 30 min)	NADES (choline chloride:lactic acid, 1:2, v/v)	HT (0.4-0.9 mg g^{-1} a));	[49]
		oleuropein (5.0–7.6 mg g^{-1} a));	
		vainillin (0.09–0.13 mg g^{-1} a));	
		rutin (0.2–0.7 mg g^{-1} a));	
		luteolin (0.10–0.17 mg g^{-1} a))	
HHPAE (8 % w/v, 600 MPa, 10 min)	NADES (choline chloride:lactic acid, 1:2, v/v)	HT (0.9–2.6 mg g^{-1} a));	[49]
		oleuropein (70.4–1.9 mg g^{-1} a));	
		vainillin (0.03–0.1 mg g^{-1} a));	
		rutin (0.2–0.7 mg g^{-1} a));	
		luteolin (0.06–0.12 mg g^{-1} a))	
Homogenization (33 % w/v, room temperature, 30 min)	Dimethyl sulfoxide	HT (about 0.7 mg g^{-1} b))	[17]
Two-phase olive pomace			
Bath-UAE (5 % w/v, 20–24 °C, 30 min)	50 % ethanol	HT, caffeic acid, *p*-coumaric acid, rutin, oleuropein y luteolin (sum of peaks 1.9–9.7 mg g^{-1} b))	[111]
MAE (5 % w/v, 90 °C, 5 min)	50 % ethanol	HT, caffeic acid, *p*-coumaric acid, rutin, oleuropein y luteolin c)	[111]
PLE (20 % w/v, 100 °C, 5 min, 103 psi)	50 % ethanol	HT, caffeic acid, *p*-coumaric acid, rutin, oleuropein y luteolin c)	[111]
Conventional extraction (3.5 % w/v, 85 °C, 240 min) e)	90 % ethanol	HT (49.3 mg g^{-1} a))	[18]
UAE (3.5 % w/v, 50 °C, 3 min, 20 kHz) e)	90 % ethanol	HT (55.1 mg g^{-1} a))	
MAE (3.5 % w/v, 50 °C, 5 min, 600 W) e)	90 % ethanol	HT (53.2 mg g^{-1} a))	
Maceration (7 % w/v, room temperature, 40 min, with agitation)	Water	HT (0.3 mg g^{-1} a); 2.0 mg g^{-1} d))	[153]
Conventional extraction (80 °C, 90 min)	1 M phosphoric acid	HT (1.4 mg g^{-1} b))	[154]
Centrifugation (10 000 × *g*, 10 min)	–	HT (5.2–6.5 mg g^{-1} d));	[67]
		tyrosol (0.5-0.7 mg g^{-1} d))	
MAE (3.5 % w/v, 14 min, 200 W)	80 % ethanol	HT (0.9 mg g^{-1} a));	[155]
		verbascoside (0.1 mg g^{-1} a))	
Pâté			
UAE (10 min, 25 °C)	Methanol	HT (0.6–0.9 mg g^{-1} a));	[53]
		verbascoside (0.6–1.3 mg g^{-1} a));	
		3,4-DHPEA-EDA (0.6–1.3 mg g^{-1} a))	

Table 2. Continued.

Extraction method	Extraction solvent	Main phenolic compounds	Ref.
Maceration (24 h, room temperature)	Water	HT (0.2–1.0 mg g^{-1} b)); verbascoside (0.8–1.0 mg g^{-1} b)); 3,4-DHPEA-EDA (1.0–1.2 mg g^{-1} b)); oleuropein (0.4–0.5 mg g^{-1} b))	[87]
Exhausted olive pomace			
Bath-UAE (6 % w/v, 50 min, 40 kHz)	47 % ethanol	HT (8 mg g^{-1} a)); oleuropein (2.0 mg g^{-1} a))	[15]
Soxhlet extraction (2 % w/v, 12 h)	Water	HT (4.1 mg g^{-1} a)); tyrosol (0.8 mg g^{-1} a))	[56]
Extraction aided by heating (10 % w/v, 85 °C, 90 min, agitated)	Water	HT (6.3 mg g^{-1} a)); tyrosol (1.6 mg g^{-1} a))	[14]
Bath-UAE (8.6 % w/v, 100 W, 43 min)	40 % acetone	HT (5.2 mg g^{-1} a))	[156]
Probe-UAE (11.5 % w/v, 70 % amplitude, 550 W, 12 min)	40 % acetone	HT (4.9 mg g^{-1} a))	
Probe-UAE (11.5 % w/v, 80 % amplitude, 550 W, 16 min)	Water	HT (6.4 mg g^{-1} a))	[88]
MAE (12 % w/v, 100 °C, 16 min)	Water	HT (5.9 mg g^{-1} a))	[22]
SFE (3.5 % w/v, 50 °C, 60 min, 40 MPa)	CO_2	HT (1.3 mg g^{-1} d))	[157]
Olive stone			
Soxhlet extraction (2 % w/v, 12 h)	Water	HT (0.1 mg g^{-1} a)); tyrosol (0.4 mg g^{-1} a))	[56]
Maceration (4 % w/v, 24 h, 4 °C)	80 % methanol	Nuezhenide (3.3 mg g^{-1} c))	[129]
Soxhlet extraction (50 % w/v, 12–24 h)	Hexane (12 h); methanol (24 h)	Pinoresinol (2.1 mg g^{-1} a))	[126]
Probe-UAE (10 % w/v, 30 min, 65 Hz)	80 % methanol + 1 N NaOH	Pinoresinol (4.0 mg g^{-1} a))	
SFE (10 % w/v, 80 °C, 40 MPa)	CO_2	Pinoresinol (1.0 mg g^{-1} a))	
Residual olive pulp			
Bath-UAE (6 % w/v, 50 min, 40 kHz)	47 % ethanol	HT (0.4 mg g^{-1} a))	[15]

3,4-DHPEA-EDA: oleacein; HAE, homogenizer-assisted extraction; HHPAE: high hydrostatic pressure; HT, hydroxytyrosol; MAE: microwave-assisted extraction; NADES: natural deep eutectic solvents; PLE: pressurized liquid extraction; SFE: supercritical fluid extraction; TPC: total phenolic compounds; UAE: ultrasound-assisted extraction.[a)]Referred to biomass dry weight. [b)]Referred to wet biomass. [c)]Not reported. [d)]Referred to extract (or liquid fraction) dry weight. [e)]Biomass was mesh screened from the kernel.

extraction time, e.g., 240 min (solvent extraction) vs. 3 min (UAE), but it was enough to provoke physical disruption and rupture of the biomass structure to favor solubilization [105] (Tab. 2).

Different studies have demonstrated that pâté contained similar phenolic compounds to that of olive pomace, e.g., HT, verbascoside, oleuropein, and luteolin [35, 53, 87] (Tab. 2). Notably, it is an interesting source of 3,4-DHPEA-EDA (or oleacein) compared to olive pomace, especially considering that it is a wet product; i.e., in dry terms, the content will be high, but more intensive drying will be required. The technologies applied most often are maceration, centrifugation, and ultrasound, but the studies provided little kinetic and mechanistic information to select the most suitable one.

Although olive pomace suffers changes during its storage in open-air ponds and processing to obtain exhausted olive pomace, the latter biomass still presents bioactive compounds to be recovered [96]. Particularly, HT stands out in the phenolic profile [14]. Hydrothermal treatments with conventional heating and steam, Soxhlet extraction, and green technologies such as UAE, MAE, and SFE have been explored for extraction. Nonetheless, the recovery of HT varies depending on the tech-

nology used and the extraction agent (Tab. 2). For example, high concentrations of HT were obtained using water as a solvent, being co-extracted with mannitol. Both compounds share high polarity (271 g L^{-1} and 216 g L^{-1}, respectively) and similar molecular weights (154.16 g mol^{-1} and 182.17 g mol^{-1}, respectively) to be extracted with water [127].

The use of supercritical CO_2 provided lower performance for HT, probably related to its low polarity. An attempt was made to solve this problem using a polar co-solvent, such as ethanol, water, and acetone, to modulate polarity and increase the extraction efficiency [109]. Regarding the technology used, UAE and extraction using conventional heating at 85 °C showed high efficiency for HT recovery using water (6.4 mg g^{-1} and 6.3 mg g^{-1}, respectively) (Tab. 2) [88]. However, UAE required a shorter time, 13 min vs. 90 min, which means energy savings. This could be related to applying a high UAE amplitude that resulted in a high extraction efficiency, but it should be applied for a short time to avoid excessive heating and degradation [88]. If UAE is then desired to be applied industrially, the acoustic intensity (W cm^{-2}), which is connected with the acoustic amplitude, exerts an important impact on the extraction yield of phenolic compounds and should be adapted to each scale [128].

Concerning the other biomasses, olive stone was suitable for obtaining pinoresinol using maceration, UAE, and SFE with CO_2 (Tab. 2) [126]. The highest concentration of this compound was obtained using UAE and methanol (80 vol %) with NaOH (1 N), although in terms of purity, SFE was more selective. Nuezhenide was also extracted by maceration with methanol (80 vol %) [129], while novel trilignols were determined in the residual pulp, which can be the basis of new studies [15].

5.4 Examples of Application of Technologies for Extraction of Triterpenic Acids

Triterpenic acids have more apolar characteristics than HT and mannitol. Generally, 90–100 % ethanol, methanol, or a mixture of both are used for their extraction from olive by-products [17, 18]. These solvents have been combined with Soxhlet extraction, maceration, UAE, and MAE, as shown in Tab. 3. Moreover, supercritical CO_2 extraction has also been applied but focused on extracting a pâté oil rich in triterpenic acids and other bioactive compounds like phytosterols and squalene [130].

The two-phase olive pomace contains triterpenic acids [16], and these compounds are highly resistant to the storage and processing conditions in the olive pomace oil extractors [17]. They are also present in exhausted olive pomace (Tab. 3). This fact may be related to the low transfer of these substances in the oils during the production processes [131].

The extraction of these compounds from biomasses depends largely on the technology used, among other factors. The data reported in Tab. 3 indicates that the use of UAE allows the highest extraction yield for both maslinic and oleanolic acids along with HT, probably, due to a higher mass transfer rate promoted by the cavitation effect. This technology and MAE were applied to recover phenolic compounds with water and triterpenic acids from EOP using maceration with ethanol in a two-step extraction. The results suggested that a selective extraction of HT by water occurs, while the extraction of the more apolar biocompounds, triterpenic acids, was favored by ethanol. Moreover, the disruptive effect of ultrasound and microwaves on the biomass structure can also facilitate their extraction (Tab. 3) [22, 88].

Fernandez-Pastor et al. [95] compared Soxhlet extraction with MAE for the extraction of triterpenic acids from residual olive pulp using methanol and ethyl acetate as extraction solvents. The results show that MAE allows for recovering a higher content of triterpenic acids than Soxhlet and in a shorter time (Tab. 3). Regarding the extracting agent used, it was observed that the proportion of triterpenic acids (oleanolic acid/maslinic acid) obtained in both techniques using ethyl acetate and methanol was similar. It can be seen that the extraction with MAE is much faster; the extraction time is six times less than that used for the conventional Soxhlet technique. This is consistent with various studies indicating that MAE technology allows for reduced extraction times and, therefore, lower energy consumption [125]. MAE is considered an emerging technique in natural product chemistry since extractions are carried out in a short time, efficiently, and under highly controlled conditions [95].

6 Integration of the Extraction of Bioactive Compounds in the Framework of Biorefineries

The term "circular economy" refers to an economic model that aims to keep resources in use for as long as possible, reduce waste, and minimize the consumption of finite resources. It is an approach that seeks to create a closed-loop system where materials and products are reused, repaired, or recycled rather than disposed of. Based on the information compiled in this review, olive pomace and derived biomasses can be valorized to recover bioactive compounds, but the remaining extracted solids are still usable. Therefore, to align with the principles of the circular (bio)economy, these solids are new resources to be valorized considering their chemical composition. After extraction, a high amount of extractable compounds (extractives) is removed, and therefore, there remains a solid enriched in polymeric components, cellulose, hemicellulose, and lignin [132]. Therefore, in this section, potential biorefinery schemes are reviewed based on this type of biomasses and include an extraction step to recover bioactive compounds.

6.1 Biorefinery Conceptual Designs

Different studies have proposed multi-product process strategies based on biomass derived from the olive oil and olive pomace oil sectors. If these biomass are used to obtain biofuels, this type of biorefinery would be considered "second generation"; that is, the biomasses have a lignocellulosic composition and do not compete with a food use [133]. In addition to combustion and cogeneration, which are currently applied in the olive sector for these biomass by-products, there are different ways to convert biomass into bioenergy and biofuels, e.g., the

Table 3. Technologies and conditions applied to obtain triterpenic acids from oil pomace and derived biomasses.

Extraction method	Solvent	Main triterpene acids	Ref.
Two-phase olive pomace			
Agitation (10 % w/v, 1 min)	Methanol-ethanol (1:1)	Maslinic acid is more abundant than oleanolic acid (total triterpenic acid content: 2.5 mg g^{-1} [a])	[17]
MAE (3.5 % w/v, 50 °C, 600 W, 5 min) [c]	90 % ethanol	Oleanolic acid (26.3 mg g^{-1} [b]); maslinic acid (356.0 mg g^{-1} [b])	[18]
UAE (3.5 % w/v, 50 °C, 136 W, 3 min) [c]	90 % ethanol	Oleanolic acid (29.8 mg g^{-1} [b]); maslinic acid (381.2 mg g^{-1} [b])	[18]
Exhausted olive pomace			
Maceration (10 % w/v, room temperature, 150 rpm, 24 h)	Ethanol	Oleanolic acid (2.1 mg g^{-1} [b]; 27.8 mg g^{-1} [d]); maslinic acid (5.7 mg g^{-1} [b]; 75.4 mg g^{-1} [d])	[88]
Probe-UAE (80 % amplitude, 16 min) [f] + maceration (10 % w/v, room temperature, 150 rpm, 24 h)	Ethanol	Oleanolic acid (3.2 mg g^{-1} [e]; 45.6 mg g^{-1} [d]); maslinic acid (8.4 mg g^{-1} [e]; 119.7 mg g^{-1} [d])	[88]
MAE (100 °C, 16 min) [f] + maceration (10 % w/v, room temperature, 150 rpm, 24 h)	Ethanol	Oleanolic acid (3.6 mg g^{-1} [e]; 53.1 mg g^{-1} [d]); maslinic acid (9.5 mg g^{-1} [e]; 140.7 mg g^{-1} [d])	[22]
Residual olive pulp			
Soxhlet extraction (5 % w/v, 75 °C, 60 min)	Ethyl acetate	Oleanolic acid (6.4–8.1 mg g^{-1} [b]); maslinic acid (15.5–17.1 mg g^{-1} [b])	[95]
MAE (5 % w/v, 85 °C, 4 min)	Ethyl acetate	Oleanolic acid (10.0–11.0 mg g^{-1} [b]); maslinic acid (22.6–23.8 mg g^{-1} [b])	
Patê			
SFE (40.2 °C, 43.8 MPa, 30 min)	CO_2	Oleanolic acid (1.1 mg g^{-1} [g]); maslinic acid (0.2 mg g^{-1} [g]); phytosterols (64.7 mg g^{-1} [g]); squalene (11.9 mg g^{-1} [g])	[130]

MAE: microwave-assisted extraction; SFE: supercritical fluid extraction; UAE: ultrasound-assisted extraction.[a]Referred to wet biomass. [b]Referred to biomass dry weight. [c]Biomass was mesh screened from the kernel. [d]Referred to extract dry weight. [e]Referred to extracted biomass dry weight. [f] Conditions applied to extract phenolic compounds, then maceration was used to extract triterpenic compounds from the recover extracted solid. [g]Referred to oil weight.

thermo-chemical route like gasification and pyrolysis to produce fuel gases, bio-char, and bio-oil as the main products, chemical routes like transesterification to obtain biodiesel, and the biochemical route focused on obtaining biogas rich in methane, ethanol, butanol, and hydrogen as clean fuels [3, 12, 61]. In the last route, the biomass must be pretreated to break down its complex structure and make its components more accessible for downstream processes. Pretreatment can be physical, chemical, or biological in nature, but combined driving forces are usually carried out [12, 134].

Tab. 4 shows some biorefinery platforms applied to olive pomace, exhausted olive pomace, and olive stone. In some cases, the integration of operations has been tested on a laboratory scale, while in other cases theoretical approximations have been made. Olive pomace provided phenolic-rich extracts along with fuel gas through pyrolysis [21, 135] and anaerobic digestion [136]. Alternatively, Ribeiro et al. [67] proposed wet and dry fractionation to recover phenolic compounds, a pulp fraction, and a stone-rich fraction as biofuel.

Several valorization strategies have been published concerning exhausted olive pomace (Tab. 4). For example, an approach obtained phenolic compounds using water as the solvent and then biogas through an anaerobic digestion [137]. These authors found that the extraction of phenolic compounds favored performing the anaerobic digestion at higher loading rates. This route is interesting since gasification is being implemented on an industrial scale in the olive sector [138]. Another strategy applied to this biomass was to recover phenolic compounds by aqueous extraction. The extracted solid was subjected to organosolv pretreatment and enzymatic hydrolysis to obtain lignin-

rich products and sugars [23]. Lignin is an interesting source of aromatic compounds to be obtained and exploited for different applications in the food industry, bioplastics, biofuel, lubricants, etc. [139].

Olive stone allowed the production of ethanol along with phenolic compounds and lignin through sequential acid pretreatment, organosolv pretreatment, enzymatic hydrolysis, and fermentation with *Saccharomyces cerevisiae*. From the xylose-rich liquor obtained in the first pretreatment step (based on diluted acid), furfural was chemically produced as another renewable product of the biorefinery [64]. These authors also proposed another similar scheme with good performance, but it was based on an alkaline pretreatment step [140]. These proposals released phenolic compounds in the pretreatment liquor, probably derived from the lignin fraction.

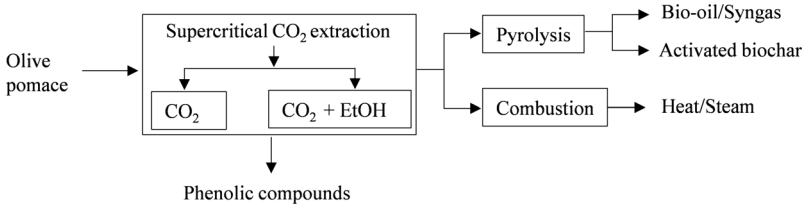
Other potential bio-based products can be produced using tailored fermentations, such as hydrogen, butanol, the sweetener xylitol, organic acids, biosurfactants, lipids, "green" plastics, etc. [12, 24, 58]. Also, the extraction of proteins has been achieved [15]. Nonetheless, these routes have not been performed in a biorefinery cascading process to also recover phenolic compounds while valorizing the rest of the components of the studied biomasses.

6.2 Sustainability and Economic Aspects

It is important to ensure that the use of biomass is sustainable and does not have negative impacts on food security, land use, and biodiversity. Therefore, developing biorefineries based on olive pomace biomasses could be a way to minimize waste. Besides the numerous works dealing with extracting phenolic compounds from olive-derived biomasses, their implementation in biorefinery cascading approaches has been little explored. As far as we know, among the "green" technologies reviewed, supercritical CO_2 extraction has been applied in

Table 4. Conversion processes and bioproducts obtained for biorefineries based on olive pomace and derived biomasses.

Biomass	Platform	Bioproducts	Scale	Conceptual design	Ref.
Olive pomace	1) Extraction + thermochemical route (pyrolysis/oxidation) 2) Extraction + thermochemical route (combustion)	1) Extract with phenolic compounds, fatty acids and squalene, syngas, bio-oil and biochar 2) Extract with phenolic compounds, fatty acids and squalene, and heat/steam	Extraction (pilot), pyrolysis (laboratory)	Integrated biorefinery based on two potential schemes, where extraction can be achieved with supercritical CO_2 or supercritical CO_2 + ethanol for the recovery of fatty acids, phenolic compounds and squalene. Then, two possibilities were considered for the extracted solid, applying a combustion that produced energy (steam and heat) and a pyrolysis that resulted in the generation of biofuels (bio-oil and syngas) and biomaterials (biochar).	[21] [135]
Two-phase olive pomace	Physical (thermo-malaxation + centrifugation) and biochemical route (anaerobic digestion)	Extract with phenolic compounds and biogas	Thermo-malaxation (pilot), anaerobic digestion (laboratory)	A biorefinery based on a second processing of the olive pomace using a three-phase decanter to obtain olive pomace oil, a liquid rich in phenolic compounds, while the solid fraction can be suitable for anaerobic digestion to produce biogas.	[137]



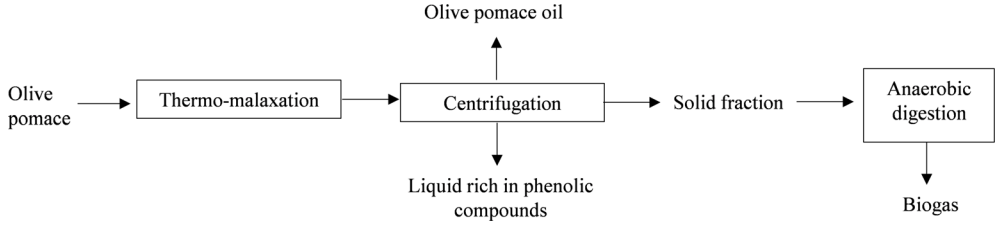


Table 4. Continued.

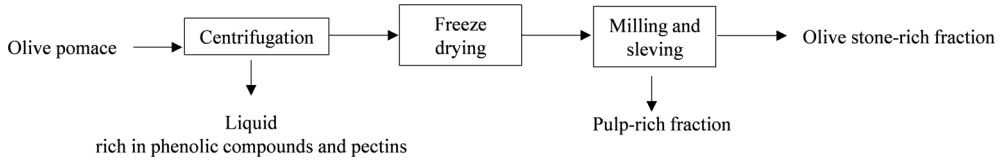
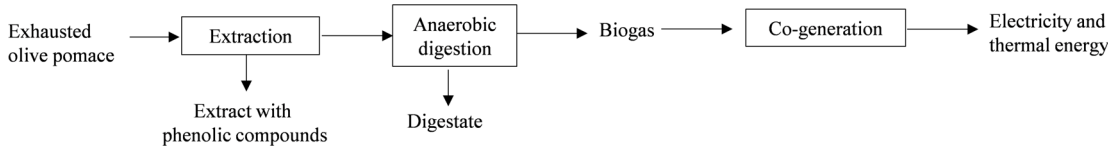
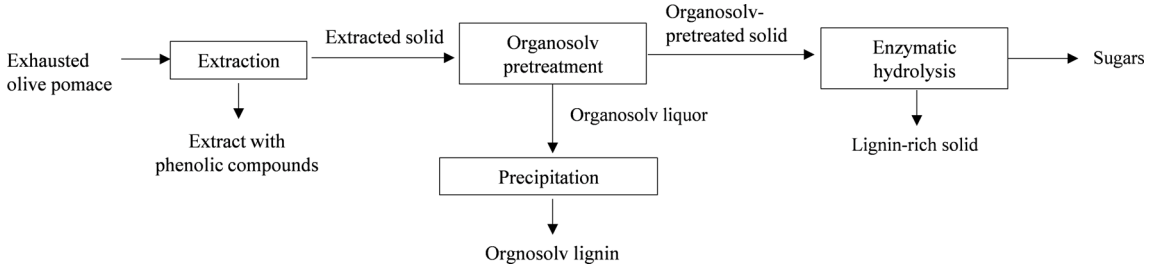
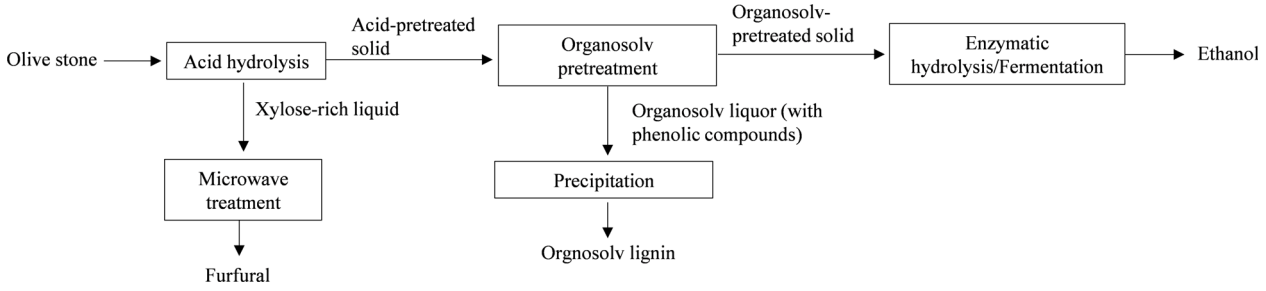
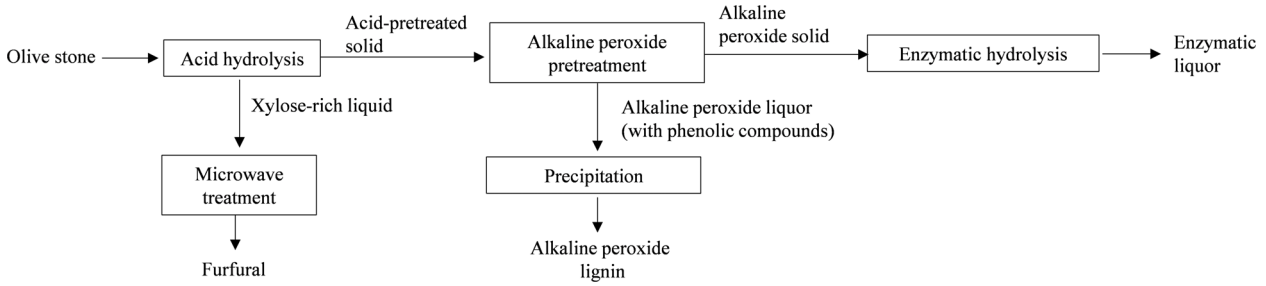
Biomass	Platform	Bioproducts	Scale	Conceptual design	Ref.
Two-phase olive pomace	Physical route: centrifugation + freeze-drying + milling + sieving	Extract rich in phenolic compounds and pectins, stone- and pulp-rich fractions	Laboratory	The process consisted of wet fractionation by centrifugation resulting in a liquid fraction rich in minerals, sugars, phenolic compounds, and pectins, while the solid fraction was dried, milled, and sieved to obtain stone- and pulp-rich fractions with high calorific value.	[67]
					
Exhausted olive pomace	Extraction + biochemical route (anaerobic digestion)	Extract rich in phenolic compounds, biogas and digestate	Laboratory	Integral valorization through the recovery of phenolic compounds by extraction with water aided by agitation and the subsequent anaerobic digestion for biogas production, which can serve to generate electricity and thermal energy by co-generation.	[137]
					
Exhausted olive pomace	Extraction + chemical (organosolv pretreatment) + biochemical route (enzymatic hydrolysis)	Extract with phenolic compounds, sugars and lignin products	Laboratory	Two-step process based on aqueous extraction to obtain a liquid extract rich in phenolic compounds and organosolv pretreatment of the extracted solid to obtain sugars and lignin.	[23]
					

Table 4. Continued.

Biomass	Platform	Bioproducts	Scale	Conceptual design	Ref.
Olive stone	Chemical (acid hydrolysis + organosolv pretreatment) + biochemical route (enzymatic hydrolysis + fermentation)	Furfural, phenolic compounds, lignin, and ethanol	Laboratory	Integrated biorefinery using a two-step fractionation process (dilute acid hydrolysis followed by organosolv) to obtain furfural from xylose via thermal treatment catalyzed by $FeCl_3$, and ethanol by simultaneous saccharification and fermentation of glucose with *Saccharomyces cerevisiae*, together with phenolic compounds and lignin.	[64]
					
Olive stone	Chemical route (acid hydrolysis pretreatment + alkaline peroxide pretreatment)	Furfural, sugars, phenolic compounds, and lignin	Laboratory	Multiproduct biorefinery based on dilute acid pretreatment to produce furfural from xylose, then alkaline peroxide pretreatment to obtain lignin, antioxidants and sugars.	[140]
					

EtOH, ethanol.

biorefinery designs [135]. The rest of the studies are based on solid-liquid extraction of phenolic compounds aided by agitation [137], centrifugation [67, 120], and heating generated by thermal conduction [14]. In any case, a high extraction yield in less time is a fundamental parameter in the field of industrial processing when looking for sustainable extractions. Among the "green" technologies used for extraction, UAE shows good performance in this sense as it reduces energy requirements and environmental impact [18, 141]. Concerning thermal-based extractions, MAE could be an efficient technology [22, 95]. Nonetheless, a recent study has shown that increasing temperature has an important environmental impact when extensive times are applied (longer than 60 min) [141]. Therefore, for MAE and PLE, temperature and time should be further optimized to increase the yield but with minimal environmental impact.

When another solvent than CO_2 is used, water is preferably applied as a "green" solvent to recover phenolic compounds. This embraces sustainable principles and is advantageous for food and pharmaceutical applications. The use of ethanol for the extraction of phenolic compounds and purification of pectins increases environmental impacts. Nonetheless, for some applications, like triterpenic acids, ethanol can be more suitable than methanol, considering its toxicity. If ethanol is recycled, the overall impacts could be reduced. So, it should be considered in the process design [141]. The impact can also be reduced using ethanol obtained from the fermentation of renewable resources instead of from the ethylene hydration process [142]. Therefore, the balance between using ethanol to increase yield and its impact should be studied for each olive resource owing to their different composition, the type of compounds extracted, and the proposed biorefinery design.

Moreover, biorefineries should be designed and operated in a way that ensures environmental and social sustainability. Although these extraction technologies are based on "green" principles, ensuring that biorefineries are sustainable is desirable. It is important to consider the entire value chain, from biomass production to product use and end-of-life management. This requires a holistic approach like an integrated life cycle sustainability assessment that considers economic, environmental, social, and organizational factors [143]. If biorefineries are coupled with novel carbon capture and storage technologies, they can even support achieving a carbon-neutral (and even negative) economy to mitigate climate change [3].

Scaling up is plausible for some of these technologies [144]. However, besides technical issues and aspects related to the process design, assessing the economic impact of the biorefinery design is also a crucial aspect in the deployment of biorefineries. This will imply calculation of the total investment capital of each biorefinery based on the technologies applied and the operational costs. The latter will be affected by regional issues, e.g., factory rents, while the breach of the material cost is much less between regions and countries [145]. As an example, Orive et al. [137] developed a techno-economic assessment of the biorefinery to valorize 1500 t/y of exhausted olive pomace by obtaining phenolic compounds, biogas, and digestate. According to the Spanish market, some assumptions concerned the selling price of the biomass, the market prices of the phenolic extracts and digestate, and savings related to electricity and thermal energy production. The conversion into phenolic compounds and biogas was economically profitable, with a net present value closer to 2 million € and a payback period of 1.7 years. An other work suggests that obtaining phenolic compounds can make ethanol-based biorefineries more profitable, especially those using lignocellulose resources [146]. However, the feasibility of the plant depended on the income from the phenolic extract sales [137].

6.3 Contributions to Sustainable Development Goals

The United Nations adopted 17 SDGs as a universal call to protect the planet and ensure that by 2030 all people enjoy peace and prosperity [147]. These goals aim to promote social, economic, and environmental sustainability at the global level. To accomplish this, the use of renewable resources is paramount. Consequently, the circular (bio)economy is linked to the SDGs because using renewable resources in all production chains contributes to fulfilling several goals [148]. In this sense, developing biorefineries using these bioresources and implementing "green" technologies and solvents would be key instruments to achieve the transition towards a circular (bio)economy with a zero-waste philosophy, also being pivotal for some SDGs.

Tab. S4 shows some SDGs to which the implementation of biorefineries based on olive pomace biomasses would contribute the most. For example, it can have numerous benefits, including reducing dependence on fossil fuels, reducing greenhouse gas emissions, creating new job opportunities, and supporting rural development, among others. The former benefits are related to SDG 7 ("Affordable and clean energy") and SDG 13 ("Climate action") since biomass has a renewable energy character and can be the basis for the production of energy, gas, and biofuels. The sustainable biorefinery concept applied to agrifood by-products/wastes is linked to SDG 12 ("Responsible consumption and production") as a way to "ensure sustainable production patterns". In terms of SDG 6 ("Clean water and sanitation") and according to [148], biorefineries can also promote increased efficiency of mass, water, and energy use through their integration in the process. It is also expected that biorefineries would increase qualified employability, going hand in hand with SDG 8 ("Decent work and economic growth"). The construction of new facilities and biorefineries would promote the industrialization of the area and foster technology innovation, contributing to SDG 9 ("Industry, innovation and infrastructure") [148]. Finally, the development of biorefineries could also contribute to interrelated SDGs, as the proper use of energy and raw materials would ensure more resources to increase well-being and living conditions.

Moreover, to ensure the achievement of the SDGs, 248 indicators have been proposed [149]. Among them, Tab. S4 also lists some indicators that can be applied to assess the real impact of implementing biorefineries on SDGs, either those proposed here or other conceptual designs and those being worked on industrially.

7 Conclusions

Numerous reports have been published which support that olive pomace and derived biomasses can be exploited as raw materials to obtain a wide range of bioactive compounds, including mannitol, xylo-oligosaccharides, pectins, phenolic compounds like HT, and triterpenic acids. However, to embrace circular (bio)economy principles, this operation should be integrated into biorefinery processes to exploit the remaining extracted solids while other products are obtained for uses such as energy, fuels, chemicals, etc. Implementing biorefineries could represent a step towards a decarbonized, circular, and more sustainable (bio)economy and support the UN's SDGs. Nevertheless, future studies should be conducted to address technical, economic, environmental, and social issues holistically.

Supporting Information

Supporting Information for this article can be found under DOI: https://doi.org/10.1002/cben.202300045. This section includes additional references to primary literature relevant for this research [158–179].

Conflicts of Interest

The authors declare no conflict of interest.

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Abbreviations

3,4-DHPEA-EDA	oleacein
DMF	multiphase decanter
HAE	homogenizer-assisted extraction
HHPAE	high hydrostatic pressure extraction
HT	hydroxytyrosol
LHW	liquid hot water
MAE	microwave-assisted extraction
NADES	natural deep eutectic solvents
N.R.	not reported
PLE	pressurized liquid extraction
SFE	supercritical fluid extraction
TFA	trifluoruoacetic acid
TPC	total phenolic compounds
UAE	ultrasound-assisted extraction
XOS	xylo-oligosaccharides

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